Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {......} EPA MRID Number 46808203

Data Requirement: PMRA Data Code:
EPA DP Barcode: D331183
OECD Data Point:
EPA Guideline: 164-1

Test material: Florasulam

End Use Product name: EF-1343 **Concentration of a.i.:** 50 g a.i./L
Formulation type: Suspension concentrate

Test material:		
Common name:		Florasulam.
Chemical name:		
	IUPAC name:	2′,6′,8-Trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide.
	CAS name:	N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide. N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy-(1,2,4)-triazolo-(1,5-c)-pyrimidine-2-sulfonamide. N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy(1,2,4)triazolo(1,5-c)pyrimidine-2-sulphonamide.
	CAS No:	145701-23-1.
	Synonyms:	DE-570; XDE-570; XR-570.
SMILES string:		C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(OC)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).

Primary Reviewer: Dan Hunt
Cambridge Environmental

Signature: Dan Hunt
Date: 07/19/07

Secondary Reviewer: Joan Harlin
Cambridge Environmental

Signature: Joan Harlin
Date: 07/19/07

QC/QA Manager: Joan Gaidos
Cambridge Environmental

Signature:
Date: 07/19/07

Final Reviewer: Cheryl Sutton
EPA Reviewer

Signature: Cheryl Sutton
Date: 1/28/2008

Company Code
Active Code
Use Site Category
EPA PC Code: 129108

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CITATION: Maycock, R. 1997. The dissipation of XDE-570 and its 5-hydroxy metabolite in soil at intervals following a single application of EF-1342, UK - 1995. Unpublished study performed by DowElanco Europe, Wantage, Oxon, United Kingdom (laboratory phase); DowElanco UK, Hitchin, Herts, United Kingdom (field management); and DowElanco Europe, Abingdon, Oxon, United Kingdom (field station), and submitted by DowElanco Europe. Report No.: GHE-P-6781. Experiment initiation April 4, 1995 (test application) and completion January 30, 1997 (final chemical analysis date; Table 3, pp. 16-17). Final report issued November 14, 1997.

EXECUTIVE SUMMARY

Soil dissipation/accumulation of florasulam (XDE-570; 2′,6′,8-trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide) under European field conditions was conducted in a bare plot of sandy clay loam soil (ADAS classification) in the United Kingdom. The experiment was carried out in accordance with Commission Directive 91/414/EEC, Annex III, Point 7.1.1, as amended by Commission Directive 95/36/EC, and in compliance with the OECD Principles of Good Laboratory Practice. Florasulam was broadcast once at a target rate of 0.015 kg a.i./ha (0.013 lb a.i./A) to a 45 x 3 m plot divided into four subplots. The timing of the application coincided with crops in adjacent fields at BBCH 31, which is typical of the timing of the test application to crops in Europe. The applied rate corresponds to 200% of the maximum proposed use rate of EF-1343 on cereals in Europe, and is three times the proposed maximum application rate (0.0045 lb ai/A or 0.005 kg ai/ha) for use in the United States. Total water input during the 372-day study period was 550 mm (21.7 inches). The historical average precipitation amount was not reported. A control plot was not established.

The application rate was not verified using application monitoring devices. Field spikes were not prepared to determine the stability of the parent and transformation products during transport and storage.

Soil samples were collected at 0, 2, 7, 14, 28, 56, 111, 195, and 372 days following the test application to a depth of 0-50 cm. Soil samples were extracted using two separate methods, an aqueous extraction and an organic extraction. In the aqueous extraction, the soil samples were extracted twice by shaking with distilled water, and the combined extracts were acidified and the analytes partitioned twice into ethyl acetate. The organic phase was then evaporated and the analytes were brought to volume with aqueous acetic acid. In the organic extraction, the soil samples were extracted twice by shaking with acetone:1% aqueous acetic acid (9:1, v:v). The combined extracts were concentrated and the analytes were purified by a non polar (C18) solid phase extraction column, followed by a strong anion exchange (SAX) Bond Elut solid phase extraction column, followed by partitioning into ethyl acetate. Extracts from both methods were analyzed for florasulam and 5-hydroxy XDE-570 (N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide) by LC/MS/MS. The LOQ was 0.05 µg/kg and the LOD was 0.01 µg/kg for both florasulam and 5-hydroxy XDE-570 for both the aqueous and organic extraction methods. The storage interval of the test samples prior to analysis was not reported.

The measured zero-time recovery of florasulam in the 0-10 cm soil layer was 6.73 µg/kg from the organic extraction (67.3% of the theoretical), decreased to 3.38 µg/kg by 2 days, ranged from 2.29 to 2.72 µg/kg from 7 to 28 days, was 0.83 µg/kg at 56 days, and was last detected above the LOQ at 0.06 µg/kg at 195 days posttreatment. Florasulam was detected in the 10-20 cm soil depth at 0.29 µg/kg at day 0, was a maximum of 1.06 µg/kg at 2 days, and was last detected at or above the LOQ at 0.05 µg/kg at 28 days posttreatment. Florasulam was detected in the 20-30 cm soil depth at 0.11-0.38 µg/kg from 0 to 14 days, in the 30-40 cm soil depth at 0.07-0.29 µg/kg from 0 to 14 days, and in the 40-50 cm soil depth at 0.08-0.28 µg/kg at 0, 2, and 14 days posttreatment. The transformation product **5-hydroxy XDE-570** was initially detected in the 0-10 cm soil depth at 0.16 µg/kg (day 0) from the organic extraction, ranged from 0.10 to 0.36 µg/kg from 2 to 14 days, was a maximum of 1.11 µg/kg at 28 days (11.1% of the applied florasulam), decreased to 0.70 µg/kg by 56 days, and was last detected above the LOQ at 0.14 µg/kg at 195 days posttreatment. 5-Hydroxy XDE-570 was detected in the 10-20 cm soil depth at a maximum of 0.20 µg/kg at day 0, ranged from 0.06 to 0.18 µg/kg from 2 to 14 days, and was last detected above the LOQ at 0.12 µg/kg at 56 days posttreatment. 5-Hydroxy XDE-570 was detected in the 20-30 cm soil depth at 0.08-0.09 µg/kg from 2 to 14 days and in the 30-40 cm depth at 0.05 µg/kg at 2 and 14 days. The reviewer-calculated half-life of 5-hydroxy XDE-570 using linear regression was 55.9 days ($r^2 = 0.9621$), based on linear regression analysis and organic-extract residue data following the maximum detection at 28 days posttreatment. The registrant-calculated DT90 was 209.5 days.

Under field conditions at the test site, florasulam had an observed DT50 of approximately 2 days (organic extraction); dissipation was biphasic, with a rapid decline to approximately 50% of the day-0 residues by 2 days posttreatment. The DT90 was 50.5 days (registrant-calculated). Residues were not present above the LOQ at the end of the 372-day study period for carryover.

The major route of dissipation of florasulam under terrestrial field conditions was transformation.

RESULTS SYNOPSIS

Location/soil type: Marcham, United Kingdom/Sandy clay loam soil (ADAS classification).
Half-life: Approximately 2 days (observed).
DT90: 50.5 days (registrant-calculated).
Major transformation products detected: 5-Hydroxy XDE-570.
Dissipation routes: Transformation.

Study Acceptability: This study is classified [to be filled in by the EFED reviewer]. No significant deviations from good scientific practices were noted. However, soils were sampled only to the 50-cm depth rather than to 90-cm, and sampling was not conducted in the two 10-cm increments below the lowest depth of leaching (50 cm). The stability of florasulam and the transformation product 5-hydroxy XDE-570 could not be determined in the test soil. The test substance was applied at three times the proposed maximum label rate in the U.S. Sampling was not performed to a sufficient depth to define leaching of the test chemical. Soil samples were excessively composited. The study was conducted at a foreign site. The soil was not adequately characterized using the USDA classification system.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: The study was conducted according to Commission Directive 91/414/EEC, Annex III, Point 7.1.1, as amended by Commission Directive 95/36/EC (p. 9). The study author also stated that the study was conducted in accordance with BBA Guideline Part IV, 4-1, December 1986 and IVA Guidelines 'Residue Studies, Part V: Studies on degradation in soil, 1993' (Appendix 1, p. 25). Deviations from USEPA Subdivision N Guideline 164-1 include:

> Storage stability data were not available for review to determine the stability of the parent and 5-hydroxy XDE-570 in the test soil during the storage interval.
>
> The test substance was applied at twice the proposed maximum label rate.
>
> Sampling was not performed to a sufficient depth to define leaching of the test chemical.
>
> Soil samples were excessively composited, providing only one sample for analysis at each sampling interval.
>
> The study author did not establish comparability between the test site soil and U.S. soils, and the soil was not adequately characterized using the USDA classification system.

COMPLIANCE: The study was conducted in compliance with OECD Principles of Good Laboratory Practice (pp. 3-3a). Signed and dated Data Confidentiality, GLP compliance and Quality Assurance statements were provided (pp. 2-4).

A. MATERIALS:

1. Test Material Florasulam (XDE-570).

Chemical Structure of the active ingredient(s): See DER Attachment 1.

Description: White crystalline solid (Appendix 3, pp. 99-100).

Storage conditions of test chemicals: The florasulam and 5-hydroxy XDE-570 analytical standards were stored refrigerated (nominally 4°C; Appendix 3, p. 77).

Physico-chemical properties of the active ingredient(s):

Paramater	Value	Comment
Chemical formula		
Molecular mass		
Water Solubility		
Vapor Pressure/Volatility		
UV Absorption		
pKa		
K_{ow}/log K_{ow}		
Stability of compound at room temperature, if provided		

2. Test site: The test site was located in Marcham, near Abingdon in Oxfordshire, United Kingdom (pp. 9-10). The test site was in an area of mixed agriculture including cereal production and the area is considered typical of a Northern European cereal growing area. The soil at the test site was classified as a sandy clay loam soil (ADAS classification; Table 3, p. 16). A pesticide use history was not reported for the test site.

Table 1: Geographic location, site description and climatic data at the study sites.

Details		Test site
Geographic coordinates	Latitude	Not reported
	Longitude	Not reported
	Province/State	Marcham
	Country	United Kingdom
	Ecoregion	Not reported
Slope Gradient		Not reported
Depth to ground water (m)		Not reported
Distance from weather station used for climatic measurements		Not reported
Indicate whether the meteorological conditions before starting or during the study were within 30 year normal levels (Yes/No). If no, provide details.		Total precipitation during the study period (measured from April 4, 1995-April 11, 1996) was 550.2 mm or 21.7 inches. The historical average precipitation amount was not reported.

Data were obtained from pp. 9-10 and Appendix 2, pp. 52-60 of the study report.

Table 2: Site usage and management history for the previous three years.

Use	Year	Test site
Crops grown	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Pesticides used	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Fertilizers used	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Cultivation methods, if provided (eg., Tillage)	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported

Prior to the test application, the plot was tilled and ring rolled (p. 10 of the study report).

3. Soils:

Table 3: Properties of the soil at the test site.

Property	Depth (cm)				
	0-10	10-20	20-30	30-40	40-50
Textural classification	Sandy clay loam[1]				
% sand	57.30	57.68	60.51	58.41	63.27
% silt	18.22	18.57	16.57	17.57	15.68
% clay	24.47	23.75	22.92	24.01	21.05
pH in water	7.7	8.0	8.0	8.0	8.1
Organic matter (%)	3.4	3.1	2.1	1.7	1.4
Total organic carbon (%)	2.0	1.8	1.2	1.0	0.8
CEC (mEq/100 g)	25.8	26.2	23.2	ND	ND
Bulk density (g/cm^3)	1.41[2]	1.42[2]	1.43[2]	1.42[2]	1.44[2]
Moisture at 1/3 atm (%)	Not reported	Not reported	Not reported	Not reported	Not reported
Taxonomic classification (e.g., ferro-humic podzol)	Calcaro-eutric cambisol				
Soil mapping unit	Not reported				

Data were obtained from pp. 9-10 and Table 3, p. 16 of the study report. Organic matter was calculated by the reviewer from percent organic carbon (% o.m. = % o.c. x 1.72). Water holding capacity was reported as 57-60% (w/w); conditions were not specified. ND = Not determined.

[1] The textural classification was reported as a sandy clay loam according to the ADAS classification; the reviewer could not determine the USDA textural classification because the two methods use different particle size ranges to classify the sand, silt, and clay fractions.

[2] Bulk density values were obtained from http://www.pedosphere.com/resources/bulkdensity/worktable_us.cfm using the site-specific sand, silt, and clay values.

B. EXPERIMENTAL DESIGN:

1. Experimental design:

Table 4: Experimental design.

Details		Test site
Duration of study		372 days
Uncropped (bare) or cropped		Bare
Control used (Yes/No)		No
No. of replications	Controls	N/A
	Treatments	One, divided into four subplots
Plot size (L x W m)	Controls	N/A
	Treatments	45 x 3 m
Distance between control plot and treated plot		N/A

Details		Test site
Distance between treated plots		N/A
Application rate(s) used (g a.i./ha)		15 g a.i./ha
Was the maximum label rate per ha used in study? (Yes/No)		No, the application rate was three times the reported maximum.
Number of applications		One
Application Date(s) (dd mm yyyy)		04/04/1995
For multiple applications, application rate at Day 0 and at each application time (mg a.i./kg soil)		0.010 mg a.i./kg[1]
Application method (eg., spraying, broadcast etc.)		Broadcast
Type of spray equipment, if used		3 m Azo boom with Lurmark SD3-F110 low drift nozzles.
Total volume of spray solution applied/plot **OR** total amount broadcasted/plot		200 L/ha
Identification and volume of carrier (e.g., water), if used		Water
Name and concentration of co-solvents, adjuvants and/or surfactants, if used		None
Indicate whether the following monthly reports were submitted: Precipitation: Average minimum and maximum air temperature: Average minimum and maximum soil temperature: Average annual frost-free periods:		 Yes, reported daily. Yes, reported daily. Yes, reported daily. No
Indicate whether the Pan evaporation data were submitted		No
Meteoro-logical conditions during application	Cloud cover	50%
	Temperature	17°C
	Humidity	62%
	Wind speed and direction	0-8 km/hr, East
	Sunlight (hr)	Not available
Pesticides used during study: Name of product/a.i conc.: Amount applied: Application method:		 Glyphosate Not reported Not reported
Supplemental irrigation used (Yes/No) If yes, provide the following details: No. of irrigation: Interval between irrigation: Amount of water added each time:		No

Details	Test site
Method of irrigation:	
Indicate whether water received through rainfall + irrigation equals the 30 year average rainfall (Yes/No)	Could not be determined.
Were the application concentrations verified?	No
Were field spikes used?	No
Good agricultural practices followed (Yes or No)	Not reported
Indicate if any abnormal climatic events occurred during the study (eg., drought, heavy rainfall, flooding, storm etc.)	The plots received 26.0 mm (1.0 inches) of rainfall on May 17-18, 1995, 28.0 mm (1.1 inches) on November 10-11, 1995, 36.0 mm (1.4 inches) on November 26-27, 1995, and 29.8 mm (1.2 inches) on December 20, 1995.
If cropped plots are used, provide the following details: Plant - Common name/variety: Details of planting: Crop maintenance:	N/A
Volatilization included in the study (Yes/No)	No
Leaching included in the study (Yes/No)	Yes
Run off included in the study (Yes/No)	No

Data were obtained from pp. 9-10; Table 3, pp. 16-17; and Appendix 2, pp. 52-60 of the study report.
1 The theoretical day-0 recovery of florasulam in the 0-10 cm depth was calculated by the reviewer using a bulk density value of 1.41 g/cm^3 and the target application rate of 0.013 lb a.i./A.

2. Application Verification: The application rate was not verified using application monitoring devices.

3. Field Spiking: Field spikes were not prepared.

4. Volatilization: Volatilization was not measured.

5. Leaching: Twenty cores (five from each subplot) were collected from the test plot to a depth of 50 cm, at 0, 2, 7, 14, 28, 56, 111, 195, and 372 days following the test application, to determine the mobility of the test substance in the soil profile (p. 10; Table 1, p. 14). Additional samples were collected at 457 and 548 days posttreatment but were not analyzed (Table 3, p. 17).

6. Run off: Run off was not studied.

7. Supplementary Study: A storage stability study using soil from the test site was not conducted.

8. Sampling:

Table 5: Soil sampling

Details	Test site
Method of sampling (random or systematic)	Systematic, along predefined lines incremented by 50 cm.
Sampling intervals	0, 2, 7, 14, 28, 56, 111, 195, 372, 457, and 548 days[1]
Method of soil collection (eg., cores)	Cores
Sampling depth	50 cm
Number of cores collected per plot	20 (5/subplot)[2]
Number of segments per core	Five
Length of soil segments (after sectioning)	10 cm
Core diameter	5 cm
Method of sample processing, if any	Composited by depth (all subplots comprising one sample) and sieved (4 mm).
Storage conditions	Frozen
Storage length (days)	Not reported

Data were obtained from p. 10 and Tables 1 and 3, pp. 14 and 17 of the study report.

1 Samples collected at 457 and 548 days were not analyzed.

2 With the exception of the 7-day sampling interval when 21 cores were collected due to sampling difficulties (5 from subplot 1, 4 from subplot 2, and 6 from subplots 3 and 4).

9. Analytical Procedures:

Number of soil samples analysed per treatment or composite sample: One, analyzed in duplicate by both analytical methods (pp. 10-11; Appendix 1, p. 40).

Extraction, clean up and concentration of soil samples:

Aqueous extraction method. Soil samples were extracted twice by shaking with distilled water, and the combined extracts were acidified and the analytes partitioned twice into ethyl acetate (p. 11). The organic phase was then evaporated and the analytes were brought to volume with aqueous acetic acid.

Organic extraction method. Additional soil samples were extracted twice by shaking with acetone:1% aqueous acetic acid (9:1, v:v) and the combined extracts were concentrated by evaporation (p. 11). Following the addition of water, the analytes were purified by a non polar (C18) solid phase extraction column, followed by a strong anion exchange (SAX) Bond Elut solid phase extraction column, followed by partitioning into ethyl acetate. Following the addition of aqueous acetic acid, the organic phase was evaporated and the remaining aqueous solution was further diluted with aqueous acetic acid.

Identification and quantification of parent compound: Both aqueous and organic extracts were analyzed for florasulam by LC/MS/MS (parameters were not reported; p. 11). Analysis employed the analytical standards Reference HD No 779/94-295 Lot 1, Sponsor Batch No

DECO 293 021 (purity 99.7%) and Reference HD No 779/94-295 Lot 3, Sponsor Batch No TSN 100381 (purity 99.7%; Appendix 3, pp. 77 and 99-100).

Identification and quantification of transformation products: Both aqueous and organic extracts were analyzed for 5-hydroxy XDE-570 (N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide; Appendix 1, p. 39) by LC/MS/MS (parameters were not reported; p. 11). Analysis employed the analytical standards Reference HD No 0060/95-295 Lot 1, Sponsor Batch No DK1-B550-16 (purity 98.7%) and Reference HD No 0060/95-295 Lot 3, Sponsor Batch No DK1-B550-16 (purity 98.1%; Appendix 3, pp. 77 and 101-102).

Detection limits (LOD, LOQ) for the parent compound in soil: The LOQ was 0.05 µg/kg and the LOD was set at 20% of the LOQ or 0.01 µg/kg (both analytical methods; p. 10; Appendix 3, p. 78).

Detection limits (LOD, LOQ) for the transformation products in soil: The LOQ was 0.05 µg/kg and the LOD was set at 20% of the LOQ or 0.01 µg/kg (both analytical methods; p. 10; Appendix 3, p. 78).

II. RESULTS AND DISCUSSION

APPLICATION MONITORS: The application rate was not verified using application monitoring devices.

2. RECOVERY FROM FIELD SPIKES: Field spikes were not prepared to determine the stability of the parent and transformation products during transport and storage.

3. MASS ACCOUNTING: A mass balance was not determined for the test site.

Table 6a. Concentration of florasulam residues expressed as µg/kg in soil from the test site – Organic extract.

Compound	**Soil depth (cm)**	**Sampling times (days)**								
		0	**2**	**7**	**14**	**28**	**56**	**111**	**195**	**372**
Florasulam	0-10	**6.73**	**3.38**	**2.34**	**2.72**	**2.29**	**0.83**	**0.20**	**0.06**	<0.05
	10-20	**0.29**	**1.06**	**0.14**	**0.62**	**0.05**	<0.05	<0.05	ND	ND
	20-30	**0.26**	**0.38**	**0.11**	**0.33**	<0.05	<0.05	NA	NA	NA
	30-40	**0.12**	**0.29**	**0.07**	**0.29**	NA	NA	NA	NA	NA
	40-50	**0.08**	**0.28**	<0.05	**0.14**	NA	NA	NA	NA	NA
5-Hydroxy XDE-570	0-10	**0.16**	**0.10**	**0.14**	**0.36**	**1.11**	**0.70**	**0.27**	**0.14**	<0.05
	10-20	**0.20**	**0.06**	**0.12**	**0.18**	<0.05	**0.12**	<0.05	<0.05	<0.05
	20-30	ND	**0.09**	**0.08**	**0.08**	ND	<0.05	NA	NA	NA
	30-40	<0.05	**0.05**[1]	<0.05	**0.05**[1]	NA	NA	NA	NA	NA
	40-50	<0.05	<0.05	<0.05	<0.05	NA	NA	NA	NA	NA

Data were obtained from Table 1, p. 14 of the study report. Total extractable and non-extractable residues were not determined. Reported residues are means of duplicate analysis unless otherwise noted; duplicate values were reported in Appendix 3, Table 1, pp. 83-85 of the study report. ND = Not detected. NA = Not analyzed.

1 One of duplicate analysis (the duplicate was <0.05 µg/kg).

Table 6b. Concentration of florasulam residues expressed as µg/kg in soil from the test site – Aqueous extract.

Compound	**Soil depth (cm)**	**Sampling times (days)**								
		0	**2**	**7**	**14**	**28**	**56**	**111**	**195**	**372**
Florasulam	0-10	**4.76**	**3.74**	**2.17**	**2.21**	**1.54**	**0.45**	**0.07**	ND	ND
	10-20	**0.15**	**0.99**	**0.07**	**0.40**	<0.05	<0.05	ND	ND	ND
	20-30	**0.24**	**0.37**	**0.08**	**0.30**	ND	ND	NA	NA	NA
	30-40	**0.13**	**0.25**	<0.05	**0.24**	NA	NA	NA	NA	NA
	40-50	**0.06**	**0.26**	<0.05	**0.12**	NA	NA	NA	NA	NA
5-Hydroxy XDE-570	0-10	**0.10**	**0.09**	**0.14**	**0.33**	**1.01**	**0.53**	**0.18**	**0.06**	<0.05
	10-20	**0.16**	<0.05	**0.08**	**0.12**	<0.05	**0.08**	<0.05	<0.05	<0.05
	20-30	ND	**0.09**	**0.07**	**0.08**	ND	<0.05	NA	NA	NA
	30-40	<0.05	<0.05	<0.05	<0.05	NA	NA	NA	NA	NA
	40-50	<0.05	<0.05	<0.05	<0.05	NA	NA	NA	NA	NA

Data were obtained from Table 2, p. 15 of the study report. Total extractable and non-extractable residues were not determined. Reported residues are means of duplicate analysis; duplicate values were reported in Appendix 3, Table 1, pp. 83-85 of the study report. ND = Not detected. NA = Not analyzed.

4. PARENT COMPOUND:

Comparison of the organic and aqueous extractable residue data sets shows that the organic extraction method recovered up to approximately 200% of the parent and up to approximately 150% of the transformation product from the soil (Tables 1-2, pp. 14-15 and Figure 1, p. 20).

Organic extractable residues.
The measured zero-time recovery of florasulam in the 0-10 cm soil layer was 6.73 µg/kg, which is 67.3% of the theoretical (reviewer-calculated based on a theoretical day-0 value of 10 µg/kg; see footnote 1 to DER Table 4; Table 1, p. 14). Following day 0, florasulam decreased to 3.38 µg/kg by 2 days, ranged from 2.29 to 2.72 µg/kg from 7 to 28 days, was 0.83 µg/kg at 56 days, and was last detected above the LOQ at 0.06 µg/kg at 195 days posttreatment. Florasulam was detected in the 10-20 cm soil depth at 0.29 µg/kg at day 0, was a maximum of 1.06 µg/kg at 2 days, and was last detected at or above the LOQ at 0.05 µg/kg at 28 days posttreatment. Florasulam was detected in the 20-30 cm soil depth at 0.11-0.38 µg/kg from 0 to 14 days, in the 30-40 cm soil depth at 0.07-0.29 µg/kg from 0 to 14 days, and in the 40-50 cm soil depth at 0.08-0.28 µg/kg at 0, 2, and 14 days posttreatment. Samples were not collected below 50 cm.

Aqueous extractable residues.
The measured zero-time recovery of florasulam in the 0-10 cm soil layer was 4.76 µg/kg, which is 47.6% of the theoretical (reviewer-calculated based on a theoretical day-0 value of 10 µg/kg; see footnote 1 to DER Table 4; Table 2, p. 15). Following day 0, florasulam decreased to 2.17-2.21 µg/kg by 7-14 days, 0.45 µg/kg by 56 days, and was last detected above the LOQ at 0.07 µg/kg at 111 days posttreatment. Florasulam was detected in the 10-20 cm soil depth at 0.15 µg/kg at day 0, was a maximum of 0.99 µg/kg at 2 days, and was last detected above the LOQ at 0.40 µg/kg at 14 days posttreatment. Florasulam was detected in the 20-30 cm soil depth at 0.08-0.37 µg/kg from 0 to 14 days, in the 30-40 cm soil depth at 0.13-0.25 µg/kg at 0, 2, and 14 days, and in the 40-50 cm soil depth at 0.06-0.26 µg/kg at 0, 2, and 14 days posttreatment. Samples were not collected below 50 cm.

HALF-LIFE: Under field conditions at the test site, florasulam had a half-life value of 30.5 days in soil ($r^2 = 0.99567$) using the organic extractable residue data set, and 19.0 days in soil ($r^2 = 0.9867$) using the aqueous extractable residue data set. The half-lives were calculated using linear regression and the equation $t_{½} = \ln 2 / k$, where k is the rate constant, and based on all available data from the 0- to 10 cm soil depth. However, the reviewer notes that the dissipation was biphasic, with a rapid decline to approximately 50% of the day-0 residues by 2 days posttreatment. The reviewer-calculated half-life of florasulam in the total soil profile (0- to 50-cm) using linear regression was 28.6 days ($r^2 = 0.9538$), calculated using the entire residue data set from the organic extraction. The DT90 was 50.5 days (registrant-calculated; pp. 12-13; Figure 2, p. 21).

5. TRANSFORMATION PRODUCTS:

Organic extractable residues.
The transformation product 5-hydroxy XDE-570 (N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide) was initially detected in the 0-10 cm soil depth at 0.16 µg/kg at day 0, ranged from 0.10 to 0.36 µg/kg from 2 to 14 days, was a maximum of 1.11 µg/kg at 28 days (11.1% of the applied florasulam; reviewer-calculated based on a theoretical day-0 florasulam value of 10 µg/kg; the 5-hydroxy XDE-570 concentration was not converted into parent equivalents), decreased to 0.70 µg/kg by 56 days, and was last detected above the LOQ at 0.14 µg/kg at 195 days posttreatment (Table 1, p. 14). 5-Hydroxy XDE-570 was detected in the 10-20 cm soil depth at a maximum of 0.20 µg/kg at day 0, ranged from 0.06 to 0.18 µg/kg from 2 to 14 days, and was last detected above the LOQ at 0.12 µg/kg at 56 days posttreatment. 5-Hydroxy XDE-570 was detected in the 20-30 cm soil depth at 0.08-0.09 µg/kg from 2 to 14 days, in the 30-40 cm depth at 0.05 µg/kg (one of duplicate analyses) at 2 and 14 days, and was not detected above the LOQ in soil below the 30-40 cm depth (Appendix 3, Table 1, pp. 83-85).

Aqueous extractable residues.
The transformation product 5-hydroxy XDE-570 was initially detected in the 0-10 cm soil depth at 0.10 µg/kg at day 0, increased to a maximum of 1.01 µg/kg by 28 days (10.1% of the applied florasulam; reviewer-calculated based on a theoretical day-0 florasulam value of 10 µg/kg; the 5-hydroxy XDE-570 concentration was not converted into parent equivalents), decreased to 0.53 µg/kg by 56 days, and was last detected above the LOQ at 0.06 µg/kg at 195 days posttreatment (Table 2, p. 15). 5-Hydroxy XDE-570 was detected in the 10-20 cm soil depth at a maximum of 0.16 µg/kg at day 0 and was 0.08-0.12 µg/kg at 7, 14, and 56 days posttreatment. 5-Hydroxy XDE-570 was detected in the 20-30 cm soil depth at 0.07-0.09 µg/kg from 2 to 14 days and was not detected above the LOQ in soil below the 20-30 cm depth.

The reviewer-calculated half-life of 5-hydroxy XDE-570 using linear regression was 55.9 days ($r^2 = 0.9621$), based on linear regression analysis and organic-extract residue data following the maximum detection at 28 days posttreatment. The study author calculated a DT50 and DT90 of 41.5 days and 209.5 days, respectively, for 5-hydroxy XDE-570 using first-order kinetics (p. 13; Figure 3, p. 22). Kinetics were performed on the residues in the 0-20 cm soil horizon from the organic extract and using the day-28 maximum concentration as the initial time-point.

Table 7: Chemical names and CAS numbers for the transformation products of florasulam.

Applicants Code Name	CAS Number	Chemical Name	Chemical Formula	Molecular Weight (g/mol)	Smiles String
5-Hydroxy XDE-570		N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide			

Data were obtained from Appendix 1, p. 39 of the study report.

6. EXTRACTABLE AND NON-EXTRACTABLE RESIDUES: Non-extractable residues were not measured.

Table 8: Dissipation routes of florasulam under field conditions.

Route of dissipation		% of applied amount	
		Organic extraction	Aqueous extraction
Accumulation (residues) in soil/ carry over		0%[1]	0%[1]
Transformation (% of transformation products)		11.1% (day 28)[2]	10.1% (day 28)[2]
Leaching, if measured (maximum depth detected)	Florasulam	40-50 cm[3]	40-50 cm[3]
	5-OH XDE-570	40-50 cm (<LOQ)[3]	40-50 cm (<LOQ)[3]
Volatilization, if measured		Not measured	Not measured
Plant uptake, if measured		N/A	N/A
Run off, if measured		Not measured	Not measured
Total			

Data were obtained from Tables 1-2, pp. 14-15 of the study report. Percentages of the applied values are reviewer-calculated based on the reviewer-calculated theoretical florasulam expected in the 0-10 cm soil depth at day 0, based on the target application rate (0.010 mg a.i./kg; see footnote 1 to DER Table 4).

1 Residues of florasulam (organic extract only) and 5-hydroxy XDE-570 were detected below the LOQ at the end of the 372-day study period.

2 Maximum concentration of 5-hydroxy XDE-570 during the study period.

3 Maximum depth sampled.

N/A = Not analyzed.

7. VOLATILIZATION: The concentration of applied florasulam lost through volatilization was not determined at the test site.

8. PLANT UPTAKE: N/A.

9. LEACHING: Residues of florasulam were detected at levels above the LOQ in soil from the lowest depth collected, the 40-50 cm depth, in both the organic and aqueous extracts (Tables 1-2, pp. 14-15). Residues of 5-hydroxy XDE-570 were not detected at levels above the LOQ in soil below the 20-30 cm depth, in both the organic and aqueous extracts, but were detected at or below the LOQ in all samples analyzed from the 30-40 and 40-50 cm soil horizons.

10. RUN OFF: Run off was not studied at the test site.

11. RESIDUE CARRYOVER: The DT90 value of florasulam was 50.5 days (registrant-calculated; p. 13; Figure 2, p. 21). Residues were not present at or above the LOQ at the end of the 372-day study period for carryover (Tables 1-2, pp. 14-15).

12. SUPPLEMENTARY STUDY RESULTS: A storage stability study was not conducted using soil from the test site.

III. STUDY DEFICIENCIES:

1. The stability of florasulam and the transformation product 5-hydroxy XDE-570 in the test soil could not be determined because a storage stability study (using either spiked field or spiked laboratory samples) was not conducted. Additionally, the length of storage of the test samples was not reported. Subdivision N Guidelines require that a storage stability study be conducted to determine the stability of the analytes under typical laboratory storage conditions.

2. The test substance was applied at an exaggerated target application rate of 0.015 kg a.i./ha (0.30 L/ha) or three times the proposed maximum label rate in the U.S. The reviewer notes that application at the maximum label rate is required for kinetic studies because dissipation rates can vary unpredictably at different application rates.

3. Sampling was not performed to a sufficient depth to define leaching of the test chemical. Residues of the parent were detected above the LOQ in the lowest soil depth collected, the 40-50 cm depth, and residues of the transformation product, 5-hydroxy XDE-570, were detected below the LOQ in the 40-50 cm soil depth (Tables 1-2, pp. 14-15). The USEPA Pesticide Registration Rejection Rate Analysis (reference 3) calls for sample cores to be collected to a depth of 90 cm, divided into segments and analyzed until one residue-free core is found. The study author stated that residues present at depths up to 50 cm within hours of application were observed in other study trials, and that the phenomenon was investigated in Report No. GHE-P-6718 and shown to be related to the use of the corer (p. 11).

4. Soil samples were excessively composited, providing only one sample for analysis at each sampling interval. The study author stated that the five soil cores from each of the four subplots were combined to give a single sample of 20 cores and that after sectioning into 10 cm horizons, the replicate horizons from all 20 cores were bulked, sieved, and analyzed in duplicate (pp. 10-11). Subdivision N Guidelines recommend that a minimum of three composites be generated per plot.

5. The field study was conducted in the United Kingdom. Subdivision N Guidelines require that terrestrial field dissipation studies be conducted domestically. Additionally, the textural classification was reported according to the ADAS classification (Table 3, p. 16); Subdivision N Guidelines require that the soil be adequately characterized using the USDA classification system.

IV. REVIEWER'S COMMENTS:

1. Historical average rainfall data were not reported to allow for comparison with the total water input from rainfall received during the study period. As a result, the reviewer could not determine whether the rainfall received during the study period was typical for the

region, and could not assess whether sufficient water was available to facilitate leaching of the test material.

2. The study author calculated the DT50 of florasulam using first-order kinetics (Timme Frehse software, version 2.00) and the residue data from the organic extractable 0-20 cm soil horizon (p. 11). Using this model, the author calculated a DT50 of 4.58 days and a DT90 of 50.49 days (pp. 12-13; Figure 2, p. 21). The study author stated that using soil residue data from the 0-10, 0-30, 0-40, and 0-50 cm soil horizons also gave a DT50 value of 5 days and a DT90 value ranging from 51–54 days (p. 13).

3. The reviewer noted that certain site-specific details were not reported, such as slope of the test plot, depth to groundwater and plot history. The reviewer also noted that pre-treatment soil samples did not contain background residues of the test compound (Tables 1-2, pp. 14-15).

4. The reported analytical methodology was not complete; LC/MS/MS parameters were not reported. Additionally, a method validation study was not performed.

5. Concurrent recoveries of florasulam and 5-hydroxy XDE-570 were determined for both the aqueous and organic extraction methods by fortifying control soil samples at fortification levels of 0.05, 1.0, 2.0, and 5.0 ppb (organic and aqueous extraction methods; p. 12; Appendix 3, p. 79). Mean recoveries for the aqueous extraction method across all fortification levels (± relative standard deviation) were 98 ± 5.3% for florasulam (range of 91 to 106%) and 90 ± 6.5% for 5-hydroxy XDE-570 (range of 79 to 101%), and mean recoveries for the organic extraction method across all fortification levels (± relative standard deviation) were 95 ± 9.9% for florasulam (range of 84 to 110%) and 93 ± 9.4% for 5-hydroxy XDE-570 (range of 84 to 109%; Appendix 3, pp. 79-80).

6. The study author stated that soil biomass values were consistent for the biological system (p. 13).

7. The reviewer noted an error in the product name in the study title. Based on the name given throughout the text of the study report, the reviewer reported the product name as EF-1343; the name reported in the study title was EF-1342.

V. REFERENCES:

1. U.S. Environmental Protection Agency. 1982. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Section 164-1, Terrestrial Field Dissipation Studies. Office of Pesticide and Toxic Substances, Washington, DC. EPA 540/9-82-021.

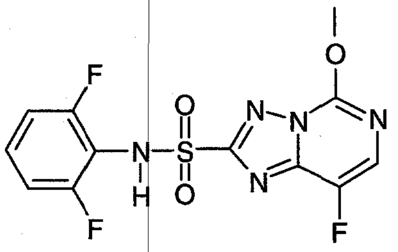
2. U.S. Environmental Protection Agency. 1993. Pesticide Registration Rejection Rate Analysis - Environmental Fate. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 738-R-93-010.

3. U.S. Environmental Protection Agency. 1989. FIFRA Accelerated Reregistration, Phase 3 Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 540/09-90-078.

Attachment 1: Structures of Parent Compound and Transformation Products

Florasulam [DE-570; XDE-570; XR-570]

IUPAC Name:	2′,6′,8-Trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide.
CAS Name:	N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide. N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy-(1,2,4)-triazolo-(1,5-c)-pyrimidine-2-sulfonamide. N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy(1,2,4)triazolo(1,5-c)pyrimidine-2-sulphonamide.
CAS Number:	145701-23-1.
SMILES String:	C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(OC)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).



Identified Compounds

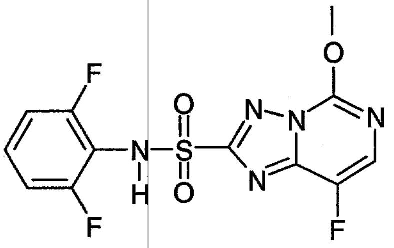
Florasulam [DE-570; XDE-570; XR-570]

IUPAC Name: 2′,6′,8-Trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide.

CAS Name: N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy-(1,2,4)-triazolo-(1,5-c)-pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy(1,2,4)triazolo(1,5-c)pyrimidine-2-sulphonamide.

CAS Number: 145701-23-1.

SMILES String: C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(OC)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).



5-Hydroxy DE-570 [5-OH; 5-OH-XDE-570; 5-OH DE-570; 5-Hydroxy-XDE-570]

IUPAC Name: Not reported.

CAS Name: N-(2,6-Difluorophenyl)-8-fluoro-5-hydroxy(1,2,4)triazolo(1,5c)pyrimidine-2-sulphonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-hydroxy(1,2,4)triazolo(1,5c)pyrimidine-2-sulfonamide.

CAS Number: Not reported.

SMILES String: C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(O)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).